Title (en)

METHOD FOR ISOLATING CAPROLACTAM FROM THE UPPER LACTAM PHASE COMPRISING THE STEPS OF 1) DISTILLATION, 2) REFINING WITH HYDROGEN IN THE PRESENCE OF A NOBEL METAL CATALYST AND 3) RECTIFICATION

Title (de)

VERFAHREN ZUR ISOLIERUNG VON CAPROLACTAM AUS DER OBEREN LACTAM-PHASE UMFASSEND DIE SCHRITTE DER 1) DESTILLATION, 2) REINIGUNG MIT WASSERSTOFF IN GEGENWART EINES EDELMETALLKATALYSATORS UND 3) REKTIFIKATION

Title (fr)

PROCÉDÉ POUR ISOLER CAPROLACTAME DE LA PHASE DE LACTAME SUPÉRIEURE COMPRENANT LES ÉTAPES DE 1) DISTILLATION, 2) RAFFINAGE AVEC D'HYDROGÈNE EN PRÉSENCE D'UN CATALYSEUR À BASE DE MÉTAL NOBLE ET 3) RECTIFICATION

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Application

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Abstract (en)

[origin: EP3660002A1] The invention relates to a method for the isolation of ε-caprolactam from the upper lactam phase obtained by separation from the sulfate phase after neutralization of the reaction mixture obtained from the Beckmann cyclohexyl oxime rearrangement with ammonia, in which the upper lactam phase is first distilled at a reduced or atmospheric pressure, by which means the water content in it is reduced to 5 to 10 % by weight, whereupon the solid phase formed by distillation is mechanically removed therefrom and the concentrated mixture thus obtained is distilled at a pressure of 0.1 to 4 kPa, whereby non-volatile components are removed therefrom. Subsequently, in the distillate thus obtained the ε-caprolactam concentration is adjusted to 30 to 70% by weight by adding at least one solvent selected from the group consisting of water, methanol, ethanol, propanol, iso-propyl alcohol, or a mixture of at least two of them and the mixture thus formed is refined by nitrogen at a temperature of 100 to 150 °C and a hydrogen pressure of 1 to 10 MPa in the presence of a supported heterogeneous catalyst which comprises at least one noble metal selected from the group consisting of Pd, Pt, Ru, whereby the multiple bonds of the impurities present are reduced. Afterwards, the raffinate thus obtained is rectified in a series of at least two rectification columns operating at a pressure of 0.05 to 4 kPa with a cumulative minimum column system efficiency of 10 theoretical plates, whereby light fractions and heavy residues are separated from the raffinate by rectification, thereby isolating ε-caprolactam therefrom.

IPC 8 full level

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